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SURVEY OF SENSITIVITY CHARACTERISTICS OF TYPICAL DELAY, IGNITER, FLASH, AND SIGNAL TYPE PYROTECHNIC COMPOSITIONS

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PICATINNY ARSENAL
DOVER, NEW JERSEY

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OBJECT

To present and interpret sensitivity data obtained for typical delay, igniter, flash, and signal type pyrotechnic compositions.

SUMMARY

Pyrotechnic compositions which have delay, igniter, flash, and signal applications were submitted for investigation with regard to their impact and friction sensitivity characteristics. Impact tests were conducted in accordance with the technique described in Technical Report FRL–TR–25 (Ref 1) except that the samples were tested as received, without performing the granulation specified. Friction tests were conducted in accordance with the procedure outlined in Picatinny Arsenal Testing Manual 7–1 (Ref 2).

The various fuels, oxidants, additives, and binders used during the study are listed below:

Fuels	Oxidants	Additives and Binders
Aluminum	Barium nitrate	Calcium fluoride
Boron	Barium chlorate	Dechlorane
Calcium	Barium chromate	Laminac resin
Calcium hydride	Barium peroxide	Polyvinyl chloride
Calcium-magnesium alloy	Manganese dioxide	Polyethylene
Magnesium	Molybdenum trioxide	Thiokol
Potassium	Sodium nitrate	Tetranitrocarbazole
Potassium borohydride	Sodium perchlorate	Nitrocellulose
Silicon	Potassium perchlorate	Vinyl-alcohol-acetate-resin
Zirconium	Strontium nitrate	
Zirconium hydride	Strontium perchlorate	
Zirconium-nickel alloy		

Extreme sensitivity to both the friction and impact tests as described in Technical Report FRL–TR–25 (Ref 1) and Testing Manual 7–1 (Ref 2) was found to be a function of the particular ingredients and the particle size of those ingredients. In general, the perchlorate containing compositions were found to be extremely sensitive to both friction and impact. The same is generally true of compositions containing finely divided fuels

such as boron, zirconium, potassium borohydride, aluminum, magnesium, and calcium and its alloys.

Organic additives in the form of binders or color intensifiers generally do not decrease and may increase sensitivity to both impact and friction when used in moderate amounts. It is concluded that extreme sensitivity to either impact or friction cannot be accurately predicted when a previously untested ingredient is incorporated into a system with known sensitivity data.

INTRODUCTION

Investigations involving the development of delay, igniter, flash, and signal compositions have always considered safety of operations as a primary obligation of the project chemist or engineer. Two procedures for measuring sensitivity make use of the Picatinny Arsenal impact test apparatus described in Technical Report FRL–TR–25 (Ref 1) and the friction pendulum apparatus described in Technical Manual 7–1 (Ref 2). The results obtained by using both of these devices enable the project chemist or engineer to plan the proper approach to the manufacture of pyrotechnic compositions and end items.

The current survey has been conducted to enable project chemists and engineers to procure sensitivity information with a minimum of expense and a maximum of efficiency. Where possible, the Picatinny Arsenal Pyrotechnics Laboratory Log Book number is indicated (Table 6, p 14), for systems described in Tables 1 through 5 (pp 9 through 13).

RESULTS AND DISCUSSION

A summary of sensitivity results is given in Tables 1 through 5. The interpretation of the results varies according to application. However, it is generally acknowledged that any pyrotechnic composition that shows any manner of combustion or explosion when subjected to the steel or fiber shoe of the friction pendulum test and/or when subjected to the 2-kilogram weight of the Picatinny Arsenal impact sensitivity apparatus must be handled with

care, particular emphasis being placed on the use of protective devices during the manufacturing process. Compositions are considered hazardous and are so designated, if reaction to the fiber shoe of the friction pendulum apparatus is evident and/or the height level of impact test reaction drops below 20 inches.

The friction pendulum test is used to determine the behavior of a sample of material exposed to a pendulum scraping across it; results are usually expressed as explosion, crackles, sparks, or no reaction or uneffected. Two stock shoes or bases to the pendulum are used, steel and a smooth-faced glossy fiber. The Picatinny Arsenal impact test is used to determine whether a reaction of any type, e.g., sparks, smoke, detonation, etc., is evident when the sample of material is subjected to the unimpeded fall of a 2-kilogram steel weight upon it. The weight is usually dropped first from a height of 12 inches. If reaction occurs, the drop height is reduced by 2 inches for the next trial. If no reaction is evident on the first try, the weight is raised 4 inches for the next trial. This raising and lowering of the weight continues until a point is reached where 10 consecutive drops are recorded with no reaction. The result is expressed in inches and is that level immediately above the level where no reaction was evident after the 10 consecutive drops.

Table 1 (p 9), entitled "Sensitivity Data for Extremely Sensitive Pyrotechnic Systems," disclosed the fact that some ingredients, such as sodium perchlorate and potassium perchlorate, contribute to the sensitivity of some systems. Both are perchlorates of alkali metals and have a history which indicates that they will combust under moderate excitation and are considered unstable materials (Ref 3). The data discloses, particularly for Systems 3, 4, 11, 12, 18, and 20 which are composed of these perchlorates and various fuels, that these oxidizing agents must be considered as the principal sensitivity contributing agent for these systems. This can be verified by comparing Systems 4 and 75, 11 and 30, 12 and 28, 18 and 38, and 20 and 17. The latter system in each pair contains non-perchlorate oxidants, and shows reduced sensitivity. It is evident that these perchlorates, when combined with powdered metals such as calcium/magnesium alloy (Systems 3, 4, and 18) or aluminum (Systems 11 and 12), produce extremely sensitive reactions when subjected to the impact and friction pendulum tests. It is also apparent that the addition of a small amount (3%) of organic additive (System 18) does not reduce the sensitivity status. In the case of sodium perchlorate as the oxidant, varying the fuels from calcium hydride (System 2) to aluminum (System 12), zirconium (System 16),

or calcium/magnesium alloy (System 19) did not materially affect the sensitivity results. With each of the systems reaction was noted with the fiber shoe of the friction pendulum apparatus. Variations in the apparatus height for the impact tests were minimal for those four systems and in each case the results must be considered as indicative of a sensitive composition.

One of the most sensitive compositions listed in Table 1 (p 9 , System 5) consisting of potassium borohydride 44% and potassium perchlorate 56% produced complete detonations when tested with either the steel or fiber shoes of the friction pendulum apparatus and provided a 7-inch result when a 2-kilogram weight was dropped on it during the impact tests. The text "Dangerous Properties of Industrial Materials," (Ref 3) refers to boron hydrides as being highly reactive and reports that heat can cause these materials to be decomposed violently. The sensitivity results obtained for System 5 bear out these warnings.

When zirconium was substituted for the potassium borohydride as a fuel (System 20), the resultant data developed from the friction pendulum and impact tests (complete detonation, steel shoe, sparks, fiber shoe, and 19-inch impact test) indicated the system was somewhat less sensitive than System 5, but still in a category where extreme care must be taken and maximum protection be provided for the operator charged with composition preparation. When atomized aluminum was substituted for the potassium borohydride (System 11), the same sensitivity level as for System 20 was reported.

It must also be noted that finely divided fuels, such as boron (1 micron, average particle diameter), atomized magnesium (23 microns, average particle diameter), and calcium hydride (4 microns, average particle diameter), when mixed with any oxidizing agent, are relatively sensitive to impact and friction. System 1 and 8, utilizing finely divided boron in both cases, had as the oxidizing agent barium chromate in the former and potassium perchlorate in the latter. Both systems were found to be extremely sensitive. When boron was used as a fuel additive in System 21 (Table 2, p 10) in the presence of finely divided atomized magnesium as the primary fuel, the same type of result was evident. When System 21 was modified to eliminate the boron (System 22, Table 2), the results indicate that the finely divided boron had in fact contributed to the comparatively sensitive nature of System 21.

As was mentioned previously small amounts of organic additives do not materially reduce and may increase the sensitivity of a system. This is further borne out when the results for Systems 18 and 19 (Table 1, p 9) and Systems 23, 24, 25, and 26 (Table 2, p 10) are examined. System 23, employing no organic binder, burned completely when subjected to the friction pendulum steel shoe, while System 24 (containing 1% Laminac binder) showed only sparking in the same test. The impact results did show greater sensitivity for System 24 than for System 23. The same comparative sensitivity was evident for Systems 19 and 18 (Table 1) and Systems 25 and 26 (Table 2), even though the latter composition in each pair contained 3% and 1% Laminac binder, respectively.

While the particle size of fuels has been observed to have a marked effect on the sensitivity of a system, no evidence was noted to support the same contention with regard to the particle size of the oxidants. Only the type of oxidant appears to have a bearing on the sensitivity results. System 29 (Table 2), employing sodium perchlorate 51% and atomized aluminum 43%, was reported as burning completely with the steel shoe and, while no reaction was evident with the fiber shoe, an impact level of 19 inches was reached, indicating that the composition is moderately sensitive. When coarse barium nitrate (147 microns average particle diameter) was substituted for the sodium perchlorate (System 28, Table 2), a marked decrease in sensitivity was noted. A radical change in the barium nitrate particle size from 147 microns to 21 microns average particle diameter (System 27, Table 2) produced only small sensitivity variations. The difference in the sensitivity data shown for Systems 33 and 34 (Table 2) further proves the fact that the type of oxidant used in a system has a marked effect on the sensitivity characteristics of that system. Compositions 33 and 34 are identical with regard to proportions of ingredients and particle size of those ingredients, the sole difference being in type of oxidant. System 33, utilizing barium nitrate as the oxidant, was reported to show sparks when the steel shoe was used in the friction pendulum test. System 34, utilizing sodium perchlorate as the oxidant, was reported to detonate completely when subjected to the same test.

Additional proof of the effect of fuel particle size was obtained when sensitivity tests were conducted on Systems 35 and 36 (Table 2). A change in the particle size of the calcium/magnesium alloy 75/25 from 100 microns (System 36) to 30 microns (System 35) produced a marked increase in sensitivity to friction from no reaction to both the steel and fiber shoes to complete burning for both shoes.

Not all pyrotechnic compositions are sensitive to impact or friction even though the perchlorates of alkali metals are used as oxidants and the fuels are finely divided. Systems 37 (Table 2, p 10) and 41 and 43 (Table 3, p 11) illustrate this possibility. In the case of System 43 (60/40/7.5 potassium perchlorate/aluminum atomized/Laminac resin), the level of organic binder (7.5 parts) was sufficient to desensitize the system. When atomized aluminum and potassium perchlorate were used without an organic binder (System 11, Table 2), complete detonations resulted with both the steel and fiber shoes. System 43 (Table 3), containing the additive calcium fluoride (30%), also showed no reaction to the steel and fiber shoes even though finely divided atomized aluminum (16 microns) and potassium perchlorate were part of the formulation. System 37 (Table 2) was found to be relatively insensitive also, possibly because barium nitrate was present in a fairly high proportion (30%). Other insensitive systems which no doubt owe their insensitivity to the diluent effects of one or more of their constituents are Systems 39, 40, 42, 44, and 45 (Table 3) and System 71 (Table 4, p 12).

Further examination of the data showed that sensitivity to friction of a particular system does not necessarily mean that that system will be sensitive to impact. The reverse is also true, in that a system sensitive to impact may not be sensitive to friction. System 47 (Table 3) was reported to show no reaction to both friction pendulum shoes and yet was reported to have a 16-inch drop test value. Conversely, System 48 (Table 3) reported to show no reaction to the impact test, combusted completely when subjected to either shoe of the friction pendulum test. System 49 (Table 3) also showed this behavior. Systems 15 (Table 1) and 37 and 38 (Table 2) may also be categorized in this manner. Systems 50, 51, 52, and 53 (Table 3); 58, 59, 60, 61, 68, and 73 (Table 4); and 75, 81, and 84 (Table 5, p 13) follow the trend normally expected, that is, when either test shows any reaction, the other test shows a similar trend.

An unusual phenomena observed during this study was the performance of compositions containing Thiokol LP–2, a polysulfide binder. In these compositions, large amounts of binder (11% and 14%, Systems 88 and 89, Table 5) did not minimize the impact sensitivity as would normally be expected. In fact, the impact values indicated a sensitivity of 14 and 13 inches, respectively, which would place these compositions in the very sensitive category even though there was no reaction when the steel shoe friction test was applied. When Thiokol was deleted from the formulation (System 30, Table 2), the sensitivity reported was 22 inches for the impact

test. This decrease in sensitivity was evident even though a more finely divided magnesium was used.

CONCLUSIONS AND RECOMMENDATIONS

Extreme sensitivity of pyrotechnic compositions to both friction and impact tests, as described in Technical Report FRL–TR–25 (Ref 1) and Testing Manual 7–1 (Ref 2), was found to be a function of the particular ingredients and the particle size of those ingredients. In general, the perchlorate containing compositions were found to be extremely sensitive to both friction and impact. The same is generally true of compositions containing finely divided fuels such as boron, zirconium, potassium borohydride, aluminum, magnesium, and calcium and its alloys.

There is a definite trend toward greater sensitivity when fuel particle size is decreased, but changing the particle size of the oxidants employed does not have any effect on sensitivity.

Organic additives in the form of color intensifiers or binders generally do not decrease and may markedly increase the sensitivity to both impact and friction when used in moderate amounts.

BLENDING AND TESTING

All compositions containing liquid binders were blended in a mortar utilizing the safety pestle in accordance with SOP–PACU–2 or SOP–PACU–3. All other compositions were blended on the Abbe Ball Mill in accordance with SOP–PACU–5.

Impact testing was conducted in accordance with P. A. Technical Report FRL–TR–25 except that the samples were tested as received instead of in the granulation specified. Friction testing was conducted in accordance with the procedure outlined in P. A. Testing Manual 7–1.

SAFETY

All operations were carried out in accordance with Ordnance Safety Manual ORDM–7–224.

REFERENCES

1. Clear, A. J., *Standard Laboratory Procedures for Sensitivity Brisance, and Stability of Explosives*, Picatinny Arsenal Technical Report FRL–TR–25, January 1961

2. McIvor, J. H., *Friction Pendulum*, Picatinny Arsenal Manual 7–1, May 1950

3. Sax, N. Irving, *Dangerous Properties of Industrial Materials*, Rheinhold Publishing Corp., pp 377–78, 990–91, 1957

TABLE 1

Sensitivity Data for Extremely Sensitive Pyrotechnic Systems

	System No.																			
	1	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**	**16**	**17**	**18**	**19**	**20**
Aluminum, atomized, 16 microns										20	24	46	10							
Boron, Amorphous, 1 micron	19							20							10					
Calcium hydride, 4 microns		38							65				53							
Calcium/magnesium alloy, 75/25, 30 microns			80	80						35								75	75	
Potassium borohydride, 20 microns					44	28	80													
Magnesium, atomized, 24 microns															5					
Zirconium, 2 microns														21		79	48.5			57
Barium chromate, 1 micron	81														85					
Molybdenum trioxide, 2 microns														26			51.5			
Potassium perchlorate, 23 microns			20		56	72	20	80	35	45	76			53						43
Sodium perchlorate, 30 microns		62										54	37			21		22	25	
Strontium perchlorate, 28 microns				20																
Laminac resin mix 4116																		3		
Friction pendulum test*																				
Steel shoe	CB	CD	CB	CD	CD	CD	CD	CB	CD	CD	CD	CD	CD	CD	CB	CD	CB	CB	CB	CD
Fibre shoe	CB	CD	CD	CD	CD	CD	CD	CB	CD	CD	CD	S	CD	CD	CB	CB	CB	CB	CB	S
Impact tests (P.A.), inches	10	15	15	10	7	9	7	8	15	18	24	19	17	8	40+	21	28	17	17	19

*CD = complete detonation

CB = complete burning

S = sparks

TABLE 2

Sensitivity Data for Flash Type Pyrotechnic Systems

	System No.																	
	21	**22**	**23**	**24**	**25**	**26**	**27**	**28**	**29**	**30**	**31**	**32**	**33**	**34**	**35**	**36**	**37**	**38**
Aluminum, atomized, 16 microns							50	50	43				31	31			40	
Aluminum, flaked, 1.4 microns													9	9				
Boron, 1 micron	4																	
Calcium, atomized, 20 microns			85	85	65	65												
Calcium/magnesium alloy, 75/25, 30 microns															75			75
Calcium/magnesium alloy, 75/25, 100 microns																75		
Magnesium, atomized, 24 microns	70	70								58	58	58						
Barium nitrate, 147 microns								50										
Barium nitrate, 21 microns							50						60				30	
Potassium perchlorate, 23 microns																	30	
Sodium perchlorate, 22 microns			15	14	35	34			57					60	25	25		
Sodium nitrate, 20 microns	30	30								42	41	39						22
Laminac resin mix 4116	2	2		1		1					1	3						3
Friction pendulum test*																		
Steel shoe	CD	S	CB	S	CD	CD	NR	NR	CB	NR	NR	S	S	CD	CB	NR	S	NR
Fibre shoe	CB	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	CB	NR	NR	NR
Impact test (P.A.), inches	20	21	20	18	16	16	31	33	19	22	20	19	28	21	17	18	40+	18

*CD = complete detonation
CB = complete burning
S = sparks
NR = no reaction

TABLE 3

Sensitivity Data for Other Pyrotechnic Systems

	System No.																	
	39	**40**	**41**	**42**	**43**	**44**	**45**	**46**	**47**	**48**	**49**	**50**	**51**	**52**	**53**	**54**	**55**	**56**
Aluminum, atomized, 16 microns			27		40	40												
Boron, 1 micron	5			5						10	5	15			14			
Calcium, atomized, 20 microns											10		25					
Magnesium, atomized, 24 microns									31.2	5	10			31.8				
Silicon, 7.6 microns		33													2			
Zirconium, 49 microns				10				35										
Zirconium hydride, 3.5 microns							40											
Barium chlorate, 26 microns									68.8									
Barium chromate, 1 micron	95			85						85	85	85	74		86			
Barium nitrate, 147 microns						50												
Barium peroxide, 6.7 microns								65										
Calcium fluoride, 5.5 microns			30															
Polyvinyl chloride, 27 microns							10											
Potassium perchlorate, 23 microns		67	43		60	10								10				53
Barium nitrate, 21 microns														68.2			68.4	
Sodium nitrate, 20 microns							47									32		
Laminac resin mix 4116					7.5		3						1			3		
Friction pendulum test*																		
Steel shoe	NR	NR	NR	NR	NR	NR	NR	NR	NR	CB	CB	CB	CB	CD	CB	CB	CD	PB
Fibre shoe	NR	NR	NR	NR	NR	NR	NR	NR	NR	CB	CB	NR	NR	NR	NR	NR	NR	NR
Impact test (P.A.), inches	40+	40+	35	40+	26	30	29	40+	16	40+	40+	11	29	27	19	21	28	24

*CD = complete detonation
CB = complete burning
PB = partial burning
NR = no reaction

TABLE 4

Sensitivity Data for Other Pyrotechnic Systems

	System No.																	
	57	**58**	**59**	**60**	**61**	**62**	**63**	**64**	**65**	**66**	**67**	**68**	**69**	**70**	**71**	**72**	**73**	**74**
Aluminum, atomized, 16 microns						35		30										
Magnesium, atomized, 24 microns		31.8							42	38	42	42	42					
Magnesium, atomized, 112 microns																60	60	66.7
Magnesium, atomized, 350 microns																		48
Magnesium/aluminum alloy, 65/35, 105 microns														37	42			
Silicon, 7.5 microns			20	20	20	10	16											
Zirconium, 49 microns	51																	
Zirconium hydride, 3.5 microns			5	7.5			15											
Zirconium/nickel alloy, 70/30, 5 microns					15													
Barium nitrate, 21 microns		68.2	50	50	50													
Dechlorane, 50 microns									7	21	5	7	7					
Laminac resin mix 4116			5	5	5	10	4									7.5	4.7	8
Polyethylene, 70 microns									7	3	9	4						
Polyvinyl chloride, 27 microns						4	5							7	16	10		2
Manganese dioxide, 4.2 microns	49																	
Nitrocellulose (in acetone)	2.6																	
Sodium nitrate, 20 microns																	28.6	42
Strontium nitrate, 30 microns						35	50	70	44	38	44	44	44	56	42	40		
Tetranitro carbazole, 3 microns		10	20	10			10											
Vinyl-alcohol-acetate resin (VAAR)												3	7					
Friction pendulum test*																		
Steel shoe	CB	CB	S	S	CB	NR	S	NR	S	NR	NR	S	C	S	NR	NR	CB	NR
Fibre shoe	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR	NR
Impact test (P.A.), inches	40+	17	18	27	21	26	37	32	16	25	18	15	19	25	40+	20	21	17

*CB = complete burning
S = sparks
C = crackles
NR = no reaction

TABLE 5

Sensitivity Data for Other Pyrotechnic Systems

	System No.														
	75	76	77	78	79	80	81	82	83	84	85	86	87	88	89
Aluminum, atomized, 16 microns										20					
Calcium, atomized, 20 microns										20					
Calcium/magnesium alloy, 75/25, 30 microns	80														
Magnesium, atomized, 112 microns					60										
Magnesium, atomized, 214 microns		15	60	45		30		29	5				29	58	
Magnesium, atomized, 350 microns		15					49		36		57	46	29		43
Barium nitrate, 21 microns		40								30					
Dechlorane, 50 microns		12													
Laminac resin mix 4116			7.5	10	7.5		8	7	10		5	9	5		
Nitrocellulose (in acetone)						1									
Polyethylene, 70 microns						5									
Potassium perchlorate, 23 microns		9			10			9		30					
Polyvinyl chloride, 27 microns			10	15	10			12				2	2		
Sodium nitrate, 20 microns	20						43		49		38	43	37	31	43
Strontium nitrate, 30 microns			40	50	40	64		43							
Thiokol LP–2 mix														11	14
Vinyl-alcohol-acetate resin (VAAR)		4													
Friction pendulum test*															
Steel shoe	CB	NR	NR	NR	NR	NR	CB	NR	NR	CD	NR	NR	NR	NR	NR
Fibre shoe	CB	–	–	–	–	–	NR	–	–	NR	–	–	–	–	–
Impact test (P.A.), inches	13	12	25	21	19	13	18	17	20	18	19	19	17	14	13

*CB = complete burning
CD = complete detonation
NR = no reaction

TABLE 6

Pyrotechnic Laboratory Reference Numbers

System No.	Pyrotechnics Laboratory Log No.	System No.	Pyrotechnics Laboratory Log No.	System No.	Pyrotechnics Laboratory Log No.
1	DP 602	31	FY 900	61	FFY 67
2	Not available	32	FY 901	62	Not available
3	PFP 699	33	PFP 54	63	FFR 27
4	Not available	34	Not available	64	PFP 685
5	PFP 716	35	Not available	65	TR 871
6	PFP 717	36	Not available	66	TR 845
7	PFP 718	37	Not available	67	TR 872
8	PFP 675	38	FY 925	68	TR 879
9	PFP 695	39	DP 478	69	TR 883
10	PFP 723	40	PFP 673	70	R–45
11	Not available	41	PFP 726	71	Not available
12	Not available	42	FW 233	72	FR 502
13	PFP 694	43	Not available	73	FY 943
14	SI 98	44	Not available	74	FY 790
15	DP 563	45	FY 623	75	FY 1008
16	Not available	46	FW 260	76	FG 491
17	DP 675	47	FG 231	77	FR 81
18	Not available	48	DP 563	78	FR 102
19	Not available	49	FW 234	79	FR 84
20	FW 116	50	DP 790	80	Not available
21	FY 771	51	FY 1088	81	FY 926
22	FY 775	52	Not available	82	FR 534
23	PFP 661	53	DP 809	83	FY 1073
24	FY 939	54	FY 903	84	Not available
25	FY 935	55	RIP–1	85	FY 845
26	FY 936	56	PFP 667	86	FY 792
27	FG 398	57	FW 244	87	FY 648
28	PFP 648	58	RIP–2	88	FY 376
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